

Appl. No. 10/817,354

Response dated 09<sup>th</sup> June 2005

Communication in reply to Notice regarding Restriction dated 10-May-05

**Listing of Claims.**

1. (Withdrawn herein) An electrical resistive device for sensing hydrogen gas, the device comprising:
  - (a) an array of titania nanotubes open at an outwardly-directed end formed by anodizing at least a portion of a titanium layer;
  - (b) a plurality of palladium clusters having been deposited atop said array of titania nanotubes; and
  - (c) said array of titania nanotubes mechanically supported by an integral support member.
2. (Withdrawn herein) The device of claim 1 wherein:
  - (a) said integral support member comprises an electrically insulative substrate layer, said top surface thereof being generally smooth; and
  - (b) said titanium layer was deposited atop said integral support member by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation.
3. (Withdrawn herein) The device of claim 1 wherein:
  - (a) said integral support member comprises an electrically insulative substrate layer; and
  - (b) said palladium clusters were deposited atop said array of nanotubes by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), electroless deposition, and laser ablation.
4. (Withdrawn herein) The device of claim 1 further comprising:
  - (a) a plurality of metal electrode-contacts deposited atop said nanotubes with said metal clusters; and
  - (b) whereby an exposure of said array of titania nanotubes to radiant ultraviolet energy in the presence of oxygen, removes at least a portion of a contaminant, if present on said titania nanotubes.
5. (Withdrawn herein) The device of claim 1 wherein:
  - (a) said substrate layer is made of an electrically insulative material, said top surface thereof being generally smooth;

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(b) a thin oxidized barrier layer is formed at the base of said array of nanotubes;  
and

(c) said integral support member further comprises a metal layer interposed between said substrate layer and a metal-oxide layer, with said oxidized barrier layer atop said metal-oxide layer.

6. (Withdrawn herein) The device of claim 1 wherein:

(a) said substrate layer is made of an electrically conductive material atop an electrically insulative base layer;

(b) a thin oxidized barrier layer is formed at the base of said array of nanotubes;  
and

(c) said integral support member further comprises an alumina nanoporous structure interposed between said electrically conductive substrate layer and a metal-oxide layer, with said oxidized barrier layer atop said metal-oxide layer.

7. (Withdrawn herein) The device of claim 1 wherein:

(a) said titanium layer is a titanium foil layer;

(b) said integral support member comprises a substrate layer comprised of a portion of said titanium foil layer that is not anodized; and

(c) said array of titania nanotubes were so formed by exposing an outwardly-directed surface of said titanium foil layer to an acidic electrolyte solution comprising a fluoride compound and an acid at a voltage selected from a range from 100 mV to 40V, for a selected time-period; and

(d) a thin oxidized barrier layer is formed at the base of said array of titania nanotubes.

8. (Withdrawn herein) The device of claim 1 wherein:

(a) said titanium layer is a doped titanium foil layer;

(b) said dopant comprises a material selected from the group consisting of: Pd, Pt, Sb, Sb<sub>2</sub>O<sub>3</sub>, In, Bi<sub>2</sub>O<sub>3</sub>, Ru, Nb, Ni, MgO, Au, Cr, Ag, Cu, N, and C;

(c) said integral support member comprises a substrate layer comprised of a portion of said doped titanium foil layer that is not anodized; and

(d) whereby an exposure of said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen, removes at least a portion of a contaminant, if present on said titania nanotubes.

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**9. (Withdrawn herein) The device of claim 1 wherein:**

- (a) said titanium layer is a titanium foil layer;
- (b) said integral support member comprises a substrate layer comprised of a portion of said titanium foil layer that is not anodized; and
- (c) said array of titania nanotubes were so formed by exposing an outwardly-directed surface of said titanium foil layer to a basic electrolyte solution at a voltage selected from a range from 100 mV to 40V, for a selected time-period within a range of 1 minute to 24 hours.

**10. (Withdrawn herein) An electrical resistive device for sensing hydrogen gas, the device comprising:**

- (a) an array of titania nanotubes comprising a dopant in an amount less than 1% by mass;
- (b) a plurality of palladium clusters having been deposited atop said array of titania nanotubes; and
- (c) said array of nanotubes mechanically supported by an integral support member.

**11. (Withdrawn herein) The device of claim 10 wherein:**

- (a) said integral support member comprises a substrate layer of an electrically insulative material;
- (b) said array of titania nanotubes were formed by anodizing at least a portion of a titanium layer comprising said dopant; and
- (c) said titanium layer comprising said dopant having been produced, prior to said anodizing, by depositing titanium and said dopant atop said integral support member by a co-deposition process selected from the group consisting of: co-sputtering, co-evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion implantation, ion plating, chemical vapor deposition, laser ablation, and thermal diffusion of said dopant into a deposited titanium matrix.

**12. (Cancel) The device of claim 11 wherein:**

- (a) a thin oxidized barrier layer is formed at the base of said array of titania nanotubes;
- (b) said integral support member further comprises a metal-oxide layer interposed between said electrically insulative substrate layer and said array of titania nanotubes, with said oxidized barrier layer atop said metal-oxide layer; and
- (c) a plurality of metal electrode-contacts are deposited atop said titania nanotubes.



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(c) said integral support member comprises a substrate layer comprised of a portion of said doped titanium foil layer that is not anodized.

**18. (Cancel) The device of claim 15 wherein:**

(a) said titanium layer is a titanium foil layer;

(b) said integral support member comprises a substrate layer comprised of a portion of said titanium foil layer that is not anodized; and

(c) said noble metal clusters were deposited atop said array of nanotubes by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), electroless deposition, and laser ablation.

**19. (Cancel) The device of claim 15 wherein:**

(a) said integral support member comprises a metal-oxide layer interposed between an electrically insulative substrate layer and said array of titania nanotubes; and

(b) said titanium layer was deposited atop said integral support member by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation.

**20. (Cancel) The device of claim 15 wherein:**

(a) said integral support member comprises a substrate layer of an electrically insulative material;

(b) said array of titania nanotubes were formed by anodizing at least a portion of a titanium layer comprising a dopant; and

(c) said titanium layer comprising said dopant having been produced, prior to said anodizing, by depositing titanium and said dopant atop said integral support member by a co-deposition process selected from the group consisting of: co-sputtering, co-evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion implantation, ion plating, chemical vapor deposition, laser ablation, and thermal diffusion of said dopant into a deposited titanium matrix.

**21. (Cancel) An electrical resistive device for sensing hydrogen gas, the device comprising:**

(a) an array of nanotubes open at an outwardly-directed end formed by anodizing at least a portion of a titanium layer comprising a dopant;

(b) said array of nanotubes having been heat treated;

(c) said array of nanotubes mechanically supported by an integral support

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member; and

(d) said titanium layer comprising said dopant having been produced, prior to said anodizing, by depositing titanium and said dopant atop said integral support member by a co-deposition process selected from the group consisting of: co-sputtering, co-evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion implantation, ion plating, chemical vapor deposition, laser ablation, and thermal diffusion of said dopant into a deposited titanium matrix.

**22. (Cancel)** The device of claim 21 adapted for use to remove a contaminant from said array of nanotubes by exposure thereof to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen; said contaminant selected from the group consisting of: liquid crude petroleum, pathogens, fungi, and proteins.

**23. (Original)** A method of producing an electrical resistive device for sensing hydrogen gas, the method comprising the steps of:

(a) forming an array of titania nanotubes open at an outwardly-directed end by anodizing at least a portion of a titanium layer;

(b) depositing a plurality of palladium clusters atop said array of titania nanotubes by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), electroless deposition, and laser ablation; and

(c) said array of titania nanotubes being mechanically supported by an integral support member.

**24. (Original)** The method of claim 23:

(a) wherein said step of forming said array comprises exposing an outwardly-directed surface of said titanium layer to an acidic electrolyte solution comprising a fluoride compound and an acid at a voltage selected from a range from 100 mV to 40V, for a selected time-period within a range of 1 minute to 24 hours; and

(b) further comprising the step of, prior to said anodizing, depositing said titanium layer atop said integral support member, which comprises an electrically insulative substrate layer, by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation.

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**25. (Original) The method of claim 23:**

(a) wherein said titanium layer comprises a titanium foil layer, and said step of forming said array comprises exposing an outwardly-directed surface of said foil layer to an acidic electrolyte solution comprising a fluoride compound, leaving a substrate layer comprised of a portion of said foil layer that is not anodized, said integral support member comprising said substrate layer; and

(b) further comprising the step of exposing said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen to remove at least a portion of a contaminant, if present on said titania nanotubes; said contaminant selected from the group consisting of: liquid crude petroleum, pathogens, fungi, and proteins.

**26. (Original). A method of producing an electrical resistive device for sensing hydrogen gas, the method comprising the steps of:**

(a) forming an array of titania nanotubes open at an outwardly-directed end by anodizing at least a portion of a titanium layer comprising a dopant in an amount less than 1% by mass;

(b) depositing a plurality of palladium clusters atop said array of titania nanotubes by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), electroless deposition, and laser ablation; and

(c) said array of titania nanotubes being mechanically supported by an integral support member.

**27. (Currently amended) The method of claim 26:**

(a) wherein said integral support member comprises a substrate layer of an electrically insulative material, said dopant comprises a material selected from the group consisting of: Pd, Pt, Sb, Sb<sub>2</sub>O<sub>3</sub>, In, Bi<sub>2</sub>O<sub>3</sub>, Ru, Nb, Ni, MgO, Au, Cr, Ag, Cu, N, and C; and

(b) further comprising the step of, prior to said anodizing, depositing titanium and said dopant atop said integral support member by a co-deposition process selected from the group consisting of: co-sputtering, co-evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion implantation, ion plating, chemical vapor deposition, laser ablation, and thermal diffusion of said dopant into a deposited titanium matrix.

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**28. (Original) The method of claim 26 wherein:**

(a) wherein said titanium layer comprises a titanium foil layer comprising said dopant, and said step of forming said array comprises exposing an outwardly-directed surface of said doped titanium foil layer to an acidic electrolyte solution comprising a fluoride compound, leaving a substrate layer comprised of a portion of said doped titanium foil layer that is not anodized, said integral support member comprising said substrate layer; and

(b) further comprising the step of exposing said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen to remove at least a portion of a contaminant, if present on said titania nanotubes; said contaminant selected from the group consisting of: liquid crude petroleum, pathogens, fungi, and proteins.

**29. (Currently amended) A method of producing an electrical resistive device for sensing hydrogen gas, the method comprising the steps of:**

(a) forming an array of titania nanotubes open at an outwardly-directed end by anodizing at least a portion of a first titanium layer;

(b) prior to said anodizing, depositing said first titanium layer atop said integral support member, which comprises an electrically insulative substrate layer, by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation; and

(c) after said step of depositing said first titanium layer and prior to said anodizing, depositing a second titanium layer, leaving a portion of said first titanium layer uncovered for said forming said array of titania nanotubes; and

~~(d) depositing a plurality of metal electrode contacts atop said titania nanotubes so-formed.~~



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**30. (Original)** A method of producing an electrical resistive device for sensing hydrogen gas, the method comprising the steps of:

- (a) forming an array of titania nanotubes open at an outwardly-directed end by anodizing at least a portion of a titanium layer;
- (b) prior to said anodizing, depositing an aluminum layer atop an electrically insulative substrate layer;
- (c) after said step of depositing said aluminum layer, depositing said titanium layer atop said aluminum layer by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation; and
- (d) after said anodizing, heat treating said array of titania nanotubes in the presence of oxygen forming a titanium-oxide layer interposed between said aluminum layer and said array of titania nanotubes.

**31. (New)** The method of claim 29:

- (a) wherein said titania nanotubes are formed to have a length greater than 600nm; and
- (b) further comprising, after said anodizing, the steps of:
  - heat treating said array of titania nanotubes in the presence of oxygen; and
  - depositing a plurality of metal electrode-contacts atop said titania nanotubes so formed.

**32. (New)** The method of claim 29:

- (a) wherein the electrical resistive device so produced is adapted for sensing hydrogen gas;
- (b) wherein said step of forming said array comprises exposing an outwardly-directed surface of said first titanium layer to an acidic electrolyte solution comprising a fluoride compound and an acid at a voltage selected from a range from 6V to 25V, for a selected time-period within a range of 1 hour to 24 hours; and
- (c) further comprising, after said step of forming said array, the step of depositing a plurality of palladium clusters atop said array of titania nanotubes.

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**33. (New)** A method of producing an electrical resistive device, the method comprising the steps of:

(a) forming an array of titania nanotubes having a length greater than 600nm and open at an outwardly-directed end by anodizing at least a portion of a titanium layer;

(b) prior to said anodizing, depositing said titanium layer atop said integral support member, which comprises an electrically insulative substrate layer, by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation; and

(c) after said anodizing, heat treating said array of titania nanotubes in the presence of oxygen.

**34. (New)** The method of claim 33 wherein:

(a) wherein the electrical resistive device so produced is adapted for sensing hydrogen gas; and

(b) further comprising the step of exposing said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen to remove at least a portion of a contaminant, if present on said titania nanotubes; said contaminant selected from the group consisting of: liquid crude petroleum, pathogens, fungi, and proteins.

**35. (New)** The method of claim 33:

(a) wherein the electrical resistive device so produced is adapted for sensing hydrogen gas;

(b) wherein said integral support member comprises a metal layer atop an electrically insulative substrate layer such that said step of depositing said titanium layer further comprises depositing said titanium layer atop said metal layer having been deposited atop said electrically insulative substrate layer; and

(c) further comprising, after said step of forming said array, the step of depositing a plurality of palladium clusters atop said array of titania nanotubes by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), electroless deposition, and laser ablation.

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**36. (New)** A method of producing an electrical resistive device, the method comprising the steps of:

- (a) forming an array of titania nanotubes having a length greater than 600nm and open at an outwardly-directed end by anodizing at least a portion of a titanium layer;
- (b) after said anodizing, heat treating said array of titania nanotubes in the presence of oxygen; and
- (c) said array of titania nanotubes being mechanically supported by an integral support member.

**37. (New)** The method of claim 36:

- (a) wherein said step of forming said array comprises exposing an outwardly-directed surface of said titanium layer to an acidic electrolyte solution comprising a fluoride compound and an acid at a voltage selected from a range from 6V to 25V, for a selected time-period within a range of 1 hour to 24 hours; and
- (b) further comprising the step of, prior to said anodizing, depositing said titanium layer atop said integral support member, which comprises an electrically insulative substrate layer, by performing a deposition process selected from the group consisting of: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition, screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), and laser ablation.

**38. (New)** The method of claim 36 further comprising the step of exposing said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen to remove at least a portion of a contaminant, if present on said titania nanotubes; said contaminant selected from the group consisting of: liquid crude petroleum, pathogens, fungi, and proteins.

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**39. (New) The method of claim 36:**

(a) wherein said titanium layer comprises a titanium foil layer, and said step of forming said array comprises exposing an outwardly-directed surface of said foil layer to an acidic electrolyte solution comprising a fluoride compound, leaving a substrate layer comprised of a portion of said foil layer that is not anodized, said integral support member comprising said substrate layer; and

(b) further comprising the step of exposing said array of titania nanotubes to radiant energy emitted within a range of frequencies from visible to ultraviolet, in the presence of oxygen to remove at least a portion of a contaminant, if present on said titania nanotubes.

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Response to Election/Restriction Communication, with Traverse

In response to the examiner's latest communication mailed 10-May-05 (due for response without extension by 10-June-05), as best as can be understood, applicants herein address their traverse as to the species sub-categorization by the examiner, and further, withdraw and amend their 22-Apr-2005 response—as was explained to the examiner during a tel. conversation the week of 23-May'05. Applicants present, here, a set of amended claims directed to a method of producing an electrical resistive device. On 14-Apr-05 the examiner had stated (copied below):

*Election/Restrictions*

1. Restriction to one of the following inventions is required under 35 U.S.C. 121:
  - I. Claims 1-22, drawn to an electrical resistive device for sensing hydrogen gas, classified in class 324, subclass 464
  - II. Claims 23-30, drawn to a method of producing an electrical resistive device for sensing hydrogen gas, classified in class 427, subclass 304, 497, and etc.

The inventions are distinct, each from the other because of the following reasons:

2. Inventions II and I are related as process of making and product made. The inventions

The examiner had further sub-characterized applicants' invention as having, within groups I and II, three patentably distinct species, as follows:

6. In the event the applicant elects group I, this group contains claims directed to the following patentably distinct species of the claimed invention:
  - (A): the species of FIGS. 1A-1E;
  - (B): the species of FIGS. 2A-2F, and
  - (C): the species of FIGS. 3A-3F.

Applicant is required under 35 U.S.C. 121 to elect a single disclosed species for prosecution on the merits to which the claims shall be restricted if no generic claim is finally

In their 22-Apr-05 communication, applicants had indicated a willingness—with traverse—to move prosecution on the merits forward by focusing initial review to those pending claims drawn to the device. The examiner has request that applicants provide support for their traverse, or further identify a subset of claims within 1 – 22 covering what the examiner has identified as species (A), FIGS 1A-1E—which the examiner asserts, and applicants agree, shows patentably distinct features from those alternatives depicted in FIGS. 2A-2F and FIGS. 3A-3F.

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Applicants note, here, that the Office has continued its practice of using technology subcategorizing, *via* species restriction requirements, on a increased basis and in manner that either forces applicants to choose to prosecute a claimset directed to a narrow target region of technology, or argue *early-on* in prosecution (that is to say, *before* the examiner has performed a search of the art) that applicants' alternatives, as pictured, do not have patentably distinct features and should be lumped together as obvious structural alternatives. This extensive use of making species restriction requirements before an examiner has searched—in light of the latest substantial increase in filing fees and per-claim fees—places an unnecessary and undue hardship on applicants who legitimately attempt to execute cost-effective strategies for pursuing patent protection of their invention with a reasonable total number of apps. pending at once.

A closer look at applicants' drawings reveals that the collective structures represented by FIGS. 1A–1E, 2A–2F, and 3A–3F, each have an 'integral support member feature' labeled 118, 38, 48 — each of the integral support member structures depicted in these figures has a 'base' substrate labeled 13(e), 21(f), 30(f). By way of example, applicants' state: "A 'base' substrate 13a-13e of an electrically insulative material such as soda glass, silica glass/silicon, quartz, ceramic, or other generally inert supportive material preferably produced such that it has a generally smooth top surface, is ultrasonically cleaned; this substrate will be of suitable thickness ... ." Applicants point out that, FIGs. 5a – 5b and 9a – 9b are SEM images of example titania nanotube array structures. Titania nanotub arrays, as explained in connection with applicants' FIG. 6 (boxes 62, 64a,b, 65): "may be produced from a titanium layer deposited atop the integral support member (step 64a, FIG. 6) using conventional film deposition techniques such as: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition (or, electroplating), screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), laser ablation, and so on," or from "an outwardly-directed surface, *e.g.*, the 'top/upper' layer of a titanium foil or doped titanium foil." To pick from among the structures represented in FIGS. 1A–1E, 2A–2F, 3A–3F is moot: Only a portion of the integral supports is different.

For handy reference, to aid in understanding, several passages of applicants' specification are reproduced next, along with corresponding figures filed by applicants:

Applicants' specification—"Summary of the Invention"—at p. 6, lines 19 – 23 states:

... The resistive devices incorporate titania nanotube array structure(s) mechanically supported by an integral support member having *one or more layers* such as an electrically insulative 'base' substrate layer, conductive foil substrate layer, metal-oxide layer(s), conductive metal layers deposited atop other layers, an alumina nanoporous structure, and so on [*emphasis added*].

Applicants' specification at p. 13, lines 8 – 16 states:

Each set of figures labeled FIGs. 1A – 1E, FIGs. 2A – 2F, and FIGs. 3A – 3F depict preferred alternative nanotube array structures at various stages of fabrication, according to the invention, in chronological, step-wise fashion. ... FIG. 4 is a schematic isometric view representative of a nanotube array structure such as is depicted by FIG. 1E, 2F, and 3F. FIGs. 5A – 5B are, respectively, top-plan view and side-view cross-sectional Field Emission Scanning Electron Microscopy (FE-SEM) images of an alternative titania nanotube array (such as those arrays schematically depicted in section, at 14b – 14e of FIGs. 1A–1E, 22b – 22f of FIGs. 2A–2F, and 31b – 31f of FIGs. 3A–3F).

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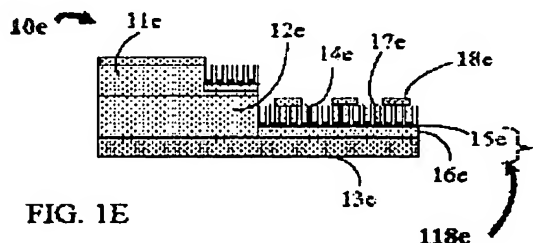


FIG. 1E

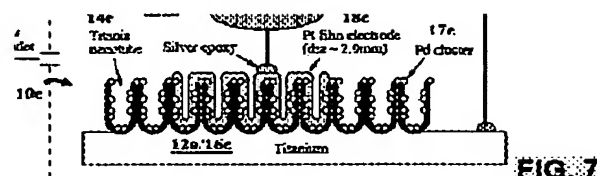


FIG. 7

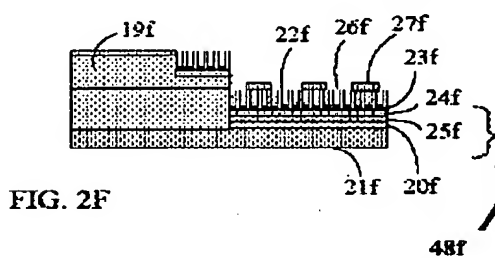


FIG. 2F

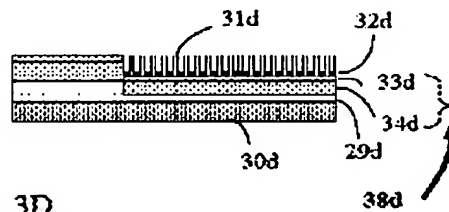
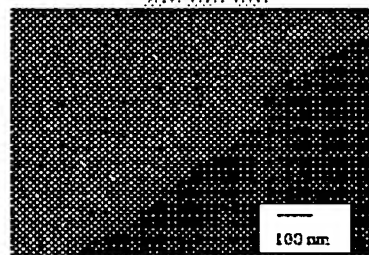


FIG. 3D

FIG. 5b



FIG. 9b



Applicants' specification at p. 13, line 22 through p. 14, line 12 states:

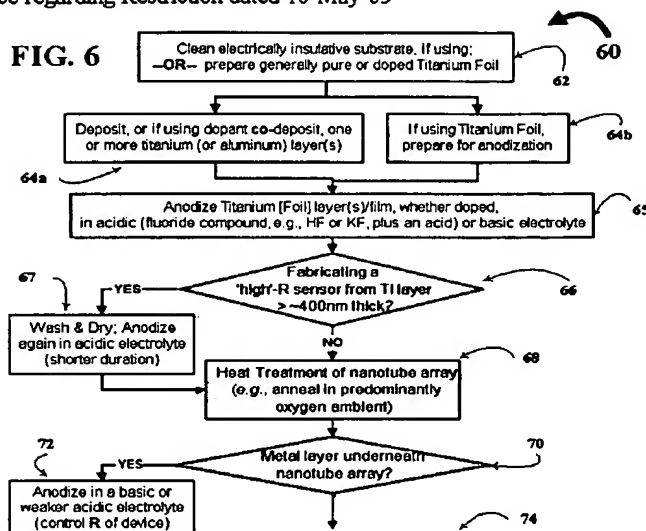
Once again as explained, in connection with reviewing the examples depicted by FIGs. 1 through 5, the method in FIG. 6, and other supporting figures, one will appreciate that: The array of titania nanotubes may be produced from a titanium layer deposited atop the integral support member (step 64a, FIG. 6) using conventional film deposition techniques such as: sputtering, evaporation using thermal energy, E-beam evaporation, ion assisted deposition, ion plating, electrodeposition (or, electroplating), screen printing, chemical vapor deposition, molecular beam epitaxy (MBE), laser ablation, and so on. The titania nanotubes may be produced from a doped titanium layer, whereby the titanium and dopant are co-deposited atop the integral support member (step 64a, FIG. 6) using convention film co-deposition techniques such as: ... Alternatively (step 64b, FIG. 6), the titania nanotubes may be formed by anodizing an outwardly-directed surface, e.g., the 'top/upper' layer of a titanium foil or doped titanium foil. Thus, as mentioned, a wide variety of integral support member configurations are contemplated for mechanically supporting the nanotube array (step 62, FIG. 6). For example, the integral support member may be composed of a metal-oxide layer interposed between a 'base' electrically insulative substrate layer and the array of titania nanotubes, with an oxidized barrier layer atop the metal-oxide layer. The integral support member may be composed of the remaining portion of Ti layer or doped Ti layer not anodized, should a Ti or doped Ti foil be used (step 64b, FIG. 6). The integral support member may also have an alumina nanoporous structure interposed between a conductive 'base' substrate layer and a metal-oxide layer.

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FIG. 6



Applicants' specification p. 15, line 18 through p. 16, line 3 describe FIGS. 1A – 1E:

The anodization of titanium 12b (FIG. 1B) in an acidic electrolyte solution forms an array of titania nanotubes 14b. The duration of anodization helps control the thickness of titanium 12b left remaining under the titania nanotubes 14b. For example, it may be desirable to make 12b extremely thin to meet desired device resistive characteristics. ... In each of FIGs. 1B – 1E the integral support has been labeled for reference, respectively, 118b-118e. ... The resulting structure is titania nanotubes 14b atop a thin layer of titanium 12b atop insulative (e.g., glass) substrate 13b. The nanotubes within the array 14b-14e are ideally well separated from each other on top as shown, while their walls are connected at the nanotube baseline by a 'barrier' Ti oxide layer 15b-15e. Barrier layer may have a thickness of ~13 nm to 20 nm, for example, or might be made thinner depending upon target resistive characteristics. ...

Applicants' specification p. 17, lines 14 – 22 describe FIGS. 2A – 2F:

... As explained in connection with FIG. 1A, an advantage of depositing a second titanium layer 19a-19f is to avoid etching titanium at the electrolyte-air interface before the completion of anodization over the base Ti layer 20a-20f. The nanotubes formed 22b-22f are oriented with open end outwardly-directed (upwardly) and a thin oxide 'barrier' layer at the base, 23b-23f. Though each nanotube as shown (ideally) interconnected at the base of the nanotube, walls of adjacent nanotubes are often in-contact somewhere between middle and bottom: This random nature to the wall thickness(es) and shapes of nanotubes formed, can be better appreciated by reviewing the side-view/sectional SEM images in FIGs. 5b, 9b.

Applicants' specification p. 15, line 18 through p. 16, line 3 describe FIGS. 3A – 3F:

... Another advantage of utilizing an underlying aluminum layer 29b is to prohibit the etching of titanium at the electrolyte-air interface in a manner as is describe above in connection with FIGs. 1A – 1E. The nanotubes, 31b-31f, are ideally well separated from each other on top while they are connected in the bottom by an oxide layer. Nanotubes are oriented with open ends outwardly-directed (at top) with a barrier layer at the bottom, 32b-32f. The as-anodized titania nanotubes are heat treated, e.g., by being annealed at 450°C in a predominantly oxygen ambient. The nanotubes 31c attain a rutile phase. A thin oxide layer 33c is formed just below barrier layer 32c. The resulting structure at 103 is titania nanotubes 31c with barrier layer 32c/ thin oxide layer 33c/ aluminum layer 29c (FIG. 3C).